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Microwave processing of Oil Sands and contribution of clay minerals

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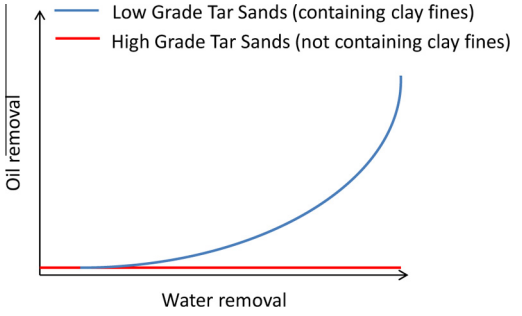
HIGHLIGHTS

- We relate the mineral composition of tar sands to their interaction with microwaves.
- Microwave treatment is feasible for Low Grade tar sands but not High Grade tar sands.
- Hydrated clay minerals are the key to successful microwave processing.
- 53% Oil removal at an energy input of 200 kW h/tonne was achieved.
- Continuous high power processing could improve oil removal and energy efficiency.

GRAPHICAL ABSTRACT



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ABSTRACT

This study establishes the feasibility of microwave heating for extracting oil from Oil Sands in ex-situ processes. Previous studies in this area have shown some potential, but have not characterised the dielectric properties of the Oil Sands used, nor related them to the mineral composition, both of which are vital if successful scale up is to be achieved. In this work the fundamental interactions of microwave energy with Oil Sands are investigated and understood for the first time, and the material characterisation related to microwave heating and oil extraction studies is carried out. It is shown that microwave heating is not feasible for extracting oil from High Grade Oil Sands due to their very low water content (<0.5%), and consequently their low dielectric loss factor. Low Grade Oil Sands can be processed, with bitumen recoveries in excess of 50% shown in this work, and energy requirements around 200 kW h/tonne. Low Grade sands contain significant clay fines and have a higher water content (~5%). This water is bound within the clay minerals, and allows temperatures in excess of 300 °C to be achieved during processing. The presence of hydrated clay minerals (predominantly kaolinite) is the key characteristic that makes Low Grade Oil Sands amenable to microwave heating. The clay minerals present in the Low Grade Oil Sands make them undesirable for conventional processing, and this study shows that microwave heating could provide a dry-processing route for recovery of bitumen from clay-containing feedstocks.

1. Introduction

The current world oil demand of 85 million bbl/day is estimated to increase to 110 million bbl/day by 2030. This demand has led to significant use of non-conventional fossil fuels such as Oil Sands. The industry is already well established and the recent multi-billion dollar investments in Canadian and Venezuelan treatment plants have resulted in the largest engineering projects in the world. According to OPEC, estimates of about 8 trillion bbl of heavy

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oil and bitumen reserves are present in various locations around the world today [1].

The interest in Oil Sand (also known as Oil Sand or Bituminous Sand) has grown considerably in the last decade, particularly in Canadian resources. Currently the daily production volume of Canadian oil from extracted bitumen is 1.5 million bbl [2]. The daily production rate is expected to treble over the next two decades to 4.5 million bbl.

Current Oil Sand processing methods include the ex-situ technique of Hot Water Separation [3,4] and a range of thermal and non-thermal in-situ techniques [4–9]. All of these processes have limitations, such as maximum depth restrictions, restriction to formations of certain dimensions and permeability, difficulty processing clay fines, high energy requirements, long processing times, requirement for the addition of hot water, gases and/or solvents, inability to process heavy oils and the need for further upgrading. Because of this, a relatively small proportion of Oil Sand reserves can be processed, and therefore there is a need for new technologies to exploit a wider range of deposits.

One technology that has been identified as having potential application to Oil Sands is microwave processing [10]. The unique ability of microwaves to heat selectively and volumetrically leads to the possibility for significant energy savings, much smaller process equipment and processes that do not require the addition of large amounts of water or caustic solution. Erdogan et al. [11] carried out dielectric property measurements on Canadian Oil Sand and suggested that they are poor candidates for microwave heating. However, other work has shown that microwave heating can be used to extract oil from Oil Sand with a high yield, and that the oil can be upgraded in the process [12]. There is clearly a need for further work to elucidate the microwave heating mechanisms of Oil Sand, and in order to understand the link between Oil Sand composition and microwave heating characteristics.

It has been well documented that aliphatic and aromatic hydrocarbons are transparent to microwaves, with dielectric loss factors <0.05 widely reported for such substances [13]. Despite their microwave transparency it has been shown that microwave heating can be used to remove oils from solid materials when there is a water phase present. Specific examples include the removal of organics from contaminated soils [14] and the removal of oil-based mud components from contaminated drill cuttings [15]. In both cases the water phase is heated selectively by microwaves and converted to steam, which acts as an in-situ mass transfer media that strips or desorbs the hydrocarbons from the host solid. In the stripping mechanism, energy may only be required to heat the water phase, rather than the entire bulk material, leading to processes that use one-fifth of the energy of their conventional heating counterparts [15]. Previous studies have highlighted the viability of continuous, industrial scale microwave processing for hydrocarbon removal where oil yields in excess of 95% were achieved [16].

The success of microwave heating for hydrocarbon removal in other applications leads to the key hypothesis posed in this study, which is that microwave heating can remove the hydrocarbon fraction (bitumen) from Oil Sands by selectively heating the water phases that are present interstitially, and within the hydrophilic clay minerals. If proven, then microwave heating could offer a step-change in Oil Sands processing as there is no need to use water or caustic solutions during the extraction, and the selective nature of the heating process may result in significantly lower energy requirements than conventional thermal processing. The potential for microwave processing is clearly significant, however there are three key questions that must be answered before the viability of the technology can be determined, and these form the objectives for this study:

2. Objectives

1. Understand the location of the water phases and interaction of microwave energy with the mineral matter within Oil Sands.
2. Determine the extent to which microwave heating can heat and remove the oil phase by selective heating of water, and the range of material properties that can be processed.
3. Understand the energy requirements to determine the relationship between power and throughput for an industrial scale system.

The quality of the hydrocarbon product is a key consideration that is not investigated in detail here. The findings of this study can be used in conjunction with a future study into hydrocarbon product quality to establish a full technology evaluation and economic analysis.

3. Methodology

3.1. Dielectric measurements

Dielectric measurements were carried out using a cavity perturbation technique [13]. This technique was chosen for its suitability for low dielectric loss materials and the ability to take high temperature readings. The system, shown in Fig. 1, consists of a cylindrical copper cavity connected to an HP 8753B vector network analyser VNA. The cavity resonates at frequencies including 910 MHz and 2.45 GHz, which are in the range allocated for microwave industrial heating.

Measurements were taken by first calculating the frequency shift and Quality factor with an empty sample-holder. The perturbation in frequency shift and Quality factor was recorded upon introduction of the Oil Sand sample, and the complex permittivity of the samples subsequently calculated using the simple perturbation equations. The process was repeated over a temperature range from 20 to 700 °C.

3.2. Microwave treatment experiments

Two different microwave experimental configurations were used in this study: a multi-mode microwave cavity was used to determine the temperature response of the Oil Sands samples to microwave heating; a single mode cavity was used to determine extraction efficiencies and energy requirements.

A multi-mode cavity was used to for the Oil Sand heating-response experiments due to its ability to heat relatively large samples. It was attached to a 3 kW microwave generator operating at 2.45 GHz and connected using segments of WR430 waveguide. The cavity dimensions were $45 \times 45 \times 70$ cm and the applied

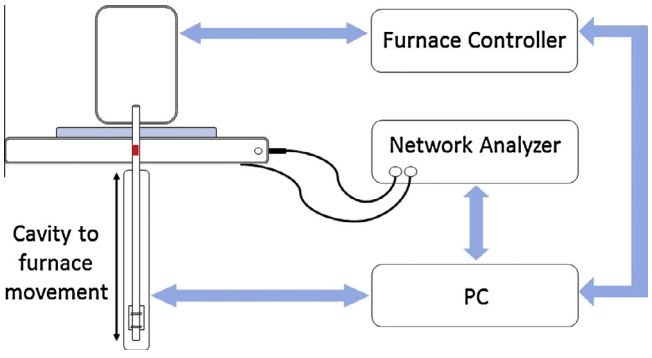

Fig. 1. Perturbation cavity.

power used was 1.5 kW. Samples weighing 150 g were placed in the cavity in a borosilicate crucible and treated for 15 s intervals. A thermocouple was inserted to measure the temperature when the microwaves were switched off. For each interval the thermocouple was inserted vertically into the beaker and the material was stirred, the highest temperature being recorded each time.

The single mode cavity set-up is shown in Fig. 2. A microwave generator with a power output of 0–6 kW was used to treat the sample at an operating frequency of 2.45 GHz. An automatic tuner was used to maximise the amount of incident power that was absorbed by the sample, and the absorbed power was recorded over time throughout the experiment. Oil Sand samples of 120 g were treated. The top of the reactor was connected to an off-gas collection system, which consisted of three condenser traps placed in cooling jackets containing dry-ice and acetone. Nitrogen was used as an inerting and carrier gas, and maintained at 5 L/min. The oil was collected from the condensers and glass tubes using dichloromethane (DCM). The collected oils were transferred to vials for weighing after evaporation of the DCM.

3.3. Materials characterisation techniques

The Dean and Stark Method (ASTM-D95) was used to determine moisture content of the Oil Sand samples. The method was repeated 5 times for a number of samples, and showed that results were reproducible to within 2.5%. Soxhlet extraction was used to determine the oil content of the Oil Sand samples, and also to extract the insoluble solid fraction from the samples for mineral analysis. The oil content results for Oil Sand presented in this study are averages of 3–5 Soxhlet extractions. X-ray diffraction (XRD) was used to identify the minerals present in the solids extracted from the Oil Sands samples. This was carried out using a Hiltonbrooks X-ray diffractometer. Gas Chromatography (GC) was used for preliminary qualitative analysis of the extracted oils. This was carried out using a Varian 1200 GC–MS system.

4. Results and discussion

4.1. Sample characterisation

The samples used in this study were sourced by Golder Associates Ltd. from the Athabasca region in Alberta, Canada. They were denoted High Grade (HG) and Low Grade (LG). Table 1 gives the oil and moisture content of the samples.

4.2. Dielectric properties

The reproducibility of the dielectric property measurements was assessed by carrying out 3 repeats on 12 samples and found to be comparable with previous measurements using this technique [17]. The dielectric properties of Low Grade and High Grade Oil Sands are shown in Fig. 3.

The average bulk dielectric loss factor of both grades varies significantly and non-linearly with temperature. The HG sample exhibits a very low loss factor, <0.03 at temperatures up to 550 °C. A different behaviour is observed for the LG sample, which shows a loss factor of 0.25–0.3 at temperatures up to 100 °C, then exhibits the same behaviour as the HG sample. The difference in loss factor below 100 °C is due to the higher water content of the LG sample, making it more microwave susceptible. The bitumen and mineral phases appear to be microwave transparent, as evidenced by the very low loss factor for both samples from 100 to 500 °C. At temperatures above 500 °C the hydrocarbon phase decomposes, leaving behind a carbonaceous deposit which is a strong microwave absorber. This behaviour has previously been documented during microwave and dielectric studies of coal and

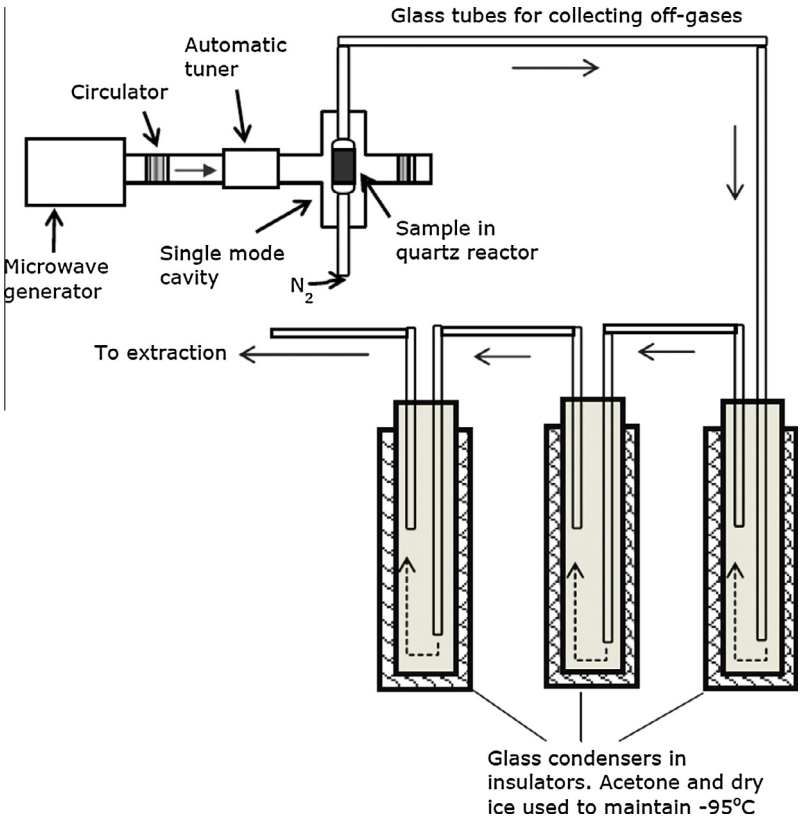

Fig. 2. Schematic of the single mode microwave treatment experiments.

Table 1
Oil and moisture content for High Grade and Low Grade Oil sand samples (based on 3–5 repeat measurements).

Sample	Oil content (wt%)	Moisture content (wt%)
Low Grade (LG)	7.1 ± 0.25	4.3 ± 0.1
High Grade (HG)	20.4 ± 0.5	0.5 ± 0.05

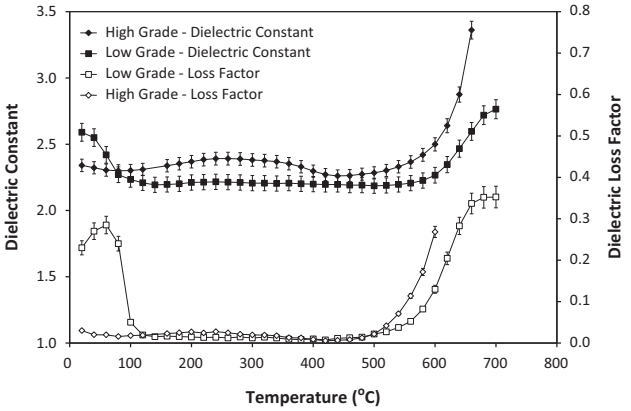

Fig. 3. Dielectric properties of HG and LG Oil Sands at 2.45 GHz.

coke, where it has been established that such structures are highly responsive to microwave energy [18].

The data shown in Fig. 3 indicates that microwave heating of the HG Oil Sand will be very challenging as it is microwave transparent up to around 500 °C. However, for the Low Grade sample Fig. 3 suggests that significant selective heating of the water phase will occur compared to the low loss hydrocarbons and mineral matter.

The location and nature of the water phase is important for two reasons. Firstly, the location affects the mobility of the water and hence the temperatures reached during heating: mass transfer limitations of the interlayer water can lead to pressure build-up in the material, resulting in superheating, whereas bulk water present in the larger pores can move to the surface unimpeded at 100 °C. Secondly, when converted to steam the water is thought to act as the primary mass transfer media for oil removal. In this study the mineral matter within the Oil Sands samples was extracted by removing the free water and the hydrocarbon phase by extensive cycles of Soxhlet extraction, followed by drying at 150 °C. The dielectric properties of these extracted solids are shown in Fig. 4.

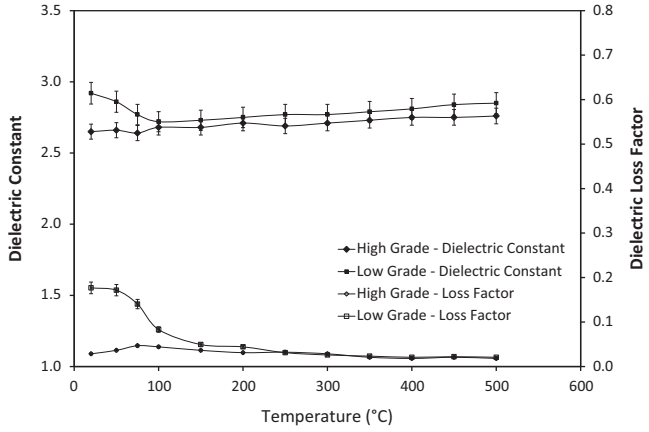

Fig. 4. Dielectric properties of the extracted solid fraction from LG and HG Oil Sands.

Fig. 4 shows that the LG Oil Sand exhibits appreciable dielectric properties at temperatures below 100 °C. The peak value of the dielectric loss is 0.18, compared with 0.3 for the bulk LG sample. The Solid Fraction within the HG Oil Sand has very low dielectric constant and loss from room temperature to 500 °C. The loss factor of the LG fraction remains higher than the HG fraction at temperatures up to 200 °C, and at higher temperatures the two fractions are comparable. The combination of water content and the nature of the mineral matter accounts for the difference in dielectric properties observed in Figs. 3 and 4. The water layer in montmorillonite clay structures has previously been shown to play an important role in the microwave heating of vermiculite [19], and is thought that the presence of hydrophilic clay minerals within the LG sample could explain the difference in dielectric properties between the different Oil Sands samples. Further mineral characterisation was undertaken to investigate this hypothesis.

4.3. Mineral characterisation

X-ray diffraction was carried out on the solid phases of the LG and HG samples, and the results are shown in Fig. 5(a) and (b). Quartz peaks are dominant in both cases, to the extent that any other structures are masked. The clay minerals were extracted from the bulk solid for further analysis. Clays are generally defined as having mean particle sizes of <2 μm [20], which is too fine to isolate using sieves alone. The extracted solid fractions were therefore sieved to separate <38 μm particles, which were then dispersed in distilled water for further grading by gravity separation. In this process, the larger particles settle more quickly than the smaller ones. The particles remaining suspended in the dispersant after 4 h are classed as <2 μm, and therefore are the clay

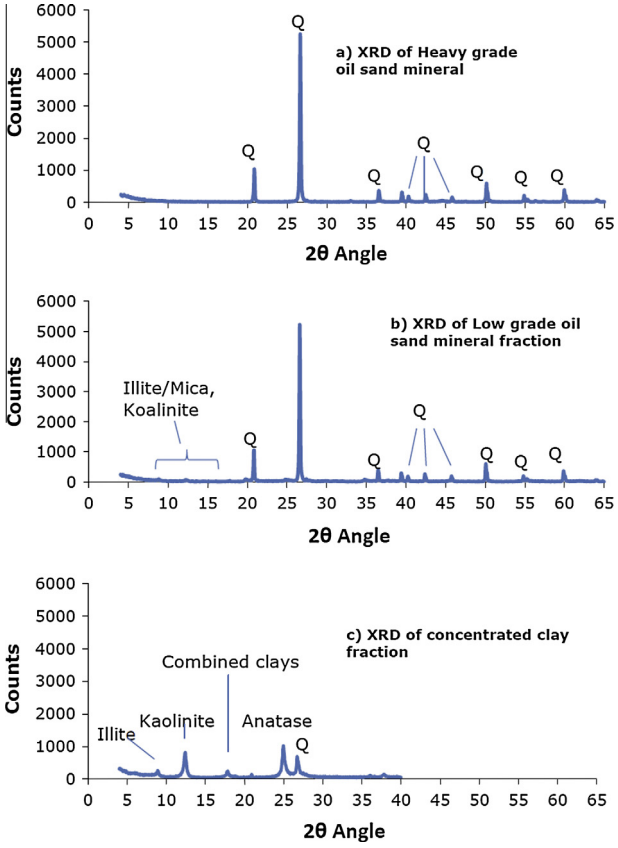

Fig. 5. XRD results for the solid phases of (a) HG Oil Sand (b) LG Oil Sand and (c) concentrated clay fraction from LG Oil Sand (Q denotes quartz).

component [21]. In the case of HG Oil Sand, no particles remained suspended, indicating the absence of clay in the sample. 7% of the solid fraction of the LG sample was collected in the suspension, and this was dried and subsequently analysed by XRD. Fig. 5(c) shows that clay minerals such as kaolinite and illite were present in the suspension obtained from the LG sample.

The solid phase of the HG Oil Sand consists entirely of quartz, which is hydrophobic. The solid phase of the LG sample is also predominantly quartz, but a significant fraction is comprised of hydrophilic mineral components (predominantly kaolinite, but also illite and other clay minerals). The hydrophilic minerals present in LG Oil Sand account for the higher water content than that of the HG material. The presence of these minerals indicates the present of interlayer water within the clay structure, in addition to free water on the surface and between grains. This interlayer water has reduced mobility compared with the free and surface water [22]. From 100 to 200 °C the loss factor of the LG sample is higher than that of the HG sample, which is also consistent with the presence of interlayer water within clay minerals and a corresponding ability to absorb microwave energy within this temperature range. At 250 °C and above the loss factor of each mineral fraction is the same, which suggests that all water is removed at 250 °C.

4.4. Thermal profiles

The dielectric property data suggests that the LG Oil Sand is a relatively good microwave absorber, whereas the HG material will be very challenging to heat. This can be validated by studying the temperature profiles over time during microwave heating. The Low and High Grade samples were studied in a multimode microwave cavity that allowed bulk temperature measurements to be obtained during processing (as detailed in Section 3.2). A power level of 1.5 kW was used, and the bulk sample temperature recorded with time. The data presented is an average of three separate experiments, with the temperature probe positioned in a different position within the sample in each case.

Fig. 6 shows that the temperature rise of the HG Oil Sand was initially of the order of 30 °C/min, but was limited to 70 °C. The LG Oil Sand exhibited a much larger temperature rise, 110 °C/min, and continued to rise beyond 250 °C. At this point the experiments were terminated due to the limitations of the apparatus used, however the data is sufficient to draw a number of key observations and conclusions, namely that the LG Oil Sand can be heated in bulk using microwaves whereas the HG sample cannot.

The water within the HG sample is not present in a large enough quantity or in the right form to enable significant microwave heating of this material. It was established in Section 3.3 that there are very few hydrophilic minerals present in the HG Oil Sand, so the water is present as free water on the surface and between grains of quartz. The water is heated selectively and freely vaporises at 100 °C. During this process, heat is transferred to the bitumen and mineral phases by conduction resulting in a bulk sample temperature of 70 °C (as observed in Fig. 6), which is the maximum temperature attainable in this case before the water is lost.

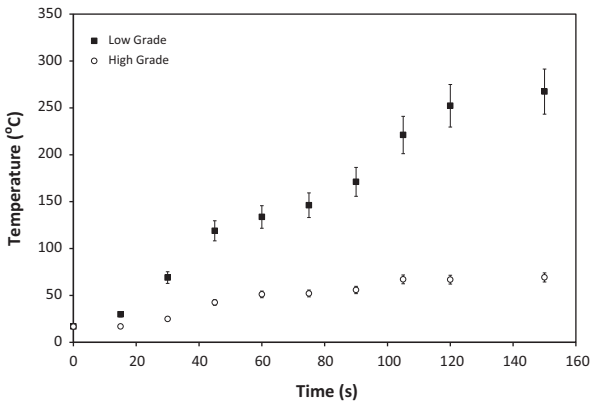

Fig. 6. Temperatures attained during microwave processing of 150 g of LG and HG Oil Sand in the multimode cavity at 1.5 kW.

The water in the LG Oil Sand is present in both free and bound forms due to the presence of clay minerals within the solid fraction. It is the latter that accounts for the difference in behaviour during microwave heating, as the bound water within the clay components of the mineral phase has reduced mobility, and so can be heated well in excess of 100 °C before it vaporises, allowing much higher bulk temperatures to be attained. The rate of temperature change in the LG sample is due to the higher water content (4.5% compared to 0.5%), meaning that much more of the applied power is absorbed than in the HG sample.

The data shown in Fig. 6 do not show the extent to which the bitumen phase can be removed, but serve to indicate the potential mechanisms by which heating takes place. If microwaves are to be used to process HG Oil Sand then clearly a mechanism must exist based on the 0.5% water that is present within these samples. Previous studies on hydrocarbon-containing materials have shown that entrainment and steam distillation can lead to hydrocarbon removal from selective heating of water using microwaves [14,15,23].

4.5. Oil recovery

Oil recovery during microwave treatment of the Oil Sands samples was carried out using a single mode cavity (detailed in Section 3.2), which enables a high power density to be attained within the heated phase, hence favouring conditions that are known to promote steam distillation and entrainment [24]. The system also allowed for accurate measurement of reflected power, from which an energy balance could be obtained, and allowed vapours to be condensed and recovered. Power levels were varied from 1 to 5 kW for up to 125 s, with a constant sample mass of 30 g. Oil and water removal was calculated based on the measured oil and water content of the feed and processed Oil Sands.

4.5.1. High Grade Oil Sand

Table 2 shows the oil and water contents of HG Oil Sand before and after treatment in the single mode cavity.

The data in Table 2 show that none of the oil was removed from the HG samples during microwave heating, but the water content was reduced by around 80%. In all cases the reflected power reached 100% after 40 s of heating, which indicates that all of the absorbent phase (water) had vaporised without removing any of the oil phase. These results lead to the conclusion that microwave heating cannot be used to remove oil from High Grade Oil Sands.

4.5.2. Low Grade Oil Sands

The degree of oil removed from 120 g of LG Oil Sand with power and time when heated in the single mode cavity is shown in Fig. 7.

Table 2
Oil and water content in the HG Oil Sand sample before and after treatment for 60 s in the single mode cavity.

Applied power (kW)	Oil content (wt%)		Water content (wt%)	
	Untreated	Treated	Untreated	Treated
1.5	20.4 ± 0.5	20.3 ± 0.3	0.5 ± 0.05	0.10 ± 0.05
3	20.4 ± 0.5	20.7 ± 0.5	0.5 ± 0.05	0.15 ± 0.05
5	20.4 ± 0.5	20.6 ± 0.2	0.5 ± 0.05	0.10 ± 0.05

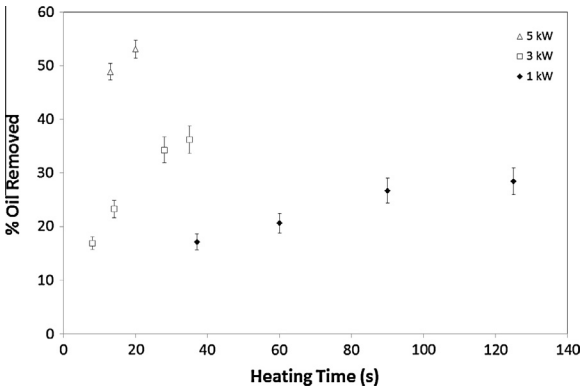

Fig. 7. Effect of heating time and power level on oil removed during microwave processing of 120 g LG Oil Sands in the single mode cavity.

Oil removal varies from 16% to 54%, depending on the processing conditions, and is in stark contrast to the behaviour of HG Oil Sands. Fig. 7 shows that oil removal increases with heating time and with applied power. At 1 kW the oil removal was 18% at 35 s, increasing to 28% at 125 s. At higher power levels the maximum observed oil removal was much higher, and in a shorter heating time, 53% in 20 s at 5 kW for example. The maximum heating time in each case was determined by the reflected power. As the water content decreases during heating, so does the dielectric loss factor (see Fig. 3), and hence the reflected power increases with time. Experiments were terminated when the reflected power reached 90% of the applied power. In varying the applied power and heating time the amount of energy absorbed by the sample was also varied. To decouple the two variables the data presented in Fig. 7 can be shown based on the energy input, which accounts for the power absorbed by the Oil Sands rather than the total applied power. This data is shown in Fig. 8.

In all cases the amount of oil removed increases with energy input, and the maximum energy absorbed by the LG Oil Sands was 770 kJ/kg. It was not possible for more energy to be absorbed due to the loss of water and corresponding increase in reflected power. Fig. 8 shows that oil removal is not solely dependent on the amount of energy absorbed. In the range of 710–770 kJ/kg, oil removal varies from 28% at 1 kW to 53% at 5 kW, which indicates that the microwave heating process is more efficient when higher powers are used at an equivalent energy input. This phenomenon has also been observed for microwave processing of oil contaminated drill cuttings [15].

The effect of power at a constant energy input is a key finding for this study, and has not been shown before for Oil Sands processing. The data in Fig. 8 imply that power density is the fundamental parameter that governs oil removal, not energy input. Power density is directly proportional to the heating rate [16]. Heating Oil Sands at a high power for a short time leads to more efficient oil removal than heating for a low power at a longer time. Higher power microwave equipment will therefore result in higher oil yields as well as allowing much higher throughputs of Oil Sand than lower power systems at equivalent energy inputs, and this finding will underpin any attempt to adopt microwave heating at a commercial scale.

There are a number of potential mechanisms that lead to the dependency on power density observed in Fig. 8. As water is the dominant microwave-absorbing phase with LG Oil Sands, it is important that the role of water in the oil removal process is understood. Fig. 9 shows how water removal varies with energy input and power, whilst Fig. 10 shows the relationship between oil and water removal.

The data in Fig. 9 shows that the water removal increases to around 90% at 300 kJ/kg, and at higher energy inputs there is a negligible change in the water content. Unlike the oil removal data shown in Fig. 8, there appears to be no effect of power on water removal in LG Oil Sands at a constant energy input.

Fig. 10 suggests that removing the first 80% of the water leads to oil removal levels of around 18%. Significant gains in oil removal are only made once 90% of the water is removed. LG Oil Sand contains both free and bound water. Around 7% of the mineral matrix is comprised of hydrophilic clay minerals, and the remainder is quartz. Free water occurs on the surface, and interstitially between grains. As it exerts its full vapour pressure it will be vaporised at 100 °C under the conditions used in this study, which is well below the temperature required to effect significant oil removal. It is thought that the threshold observed in Fig. 10 corresponds to the amount of free water present within the LG Oil Sands. Free water is heated selectively during microwave heating, and as it vaporises there is thought to be limited steam distillation or entrainment of the oil phase with the steam, resulting in low levels of oil removal. This effect was not seen in the HG Oil Sands, and this is because there was only 0.5% water (compared with 4.3% in the LG sample), meaning that the energy absorbed by the system was much lower in the case of the HG Oil Sands and therefore the oil removal was negligible. The remainder of the water within LG Oil Sands is bound water, which does not exert its full vapour pressure and will be present within the hydrophilic mineral matter at much higher temperatures. As the bound water is heated, temperatures in excess of 250 °C can be obtained (from Fig. 6), which is high enough to vaporise the lighter hydrocarbon components within the bitumen phase and also to thermally decompose some of the heavier hydrocarbons.

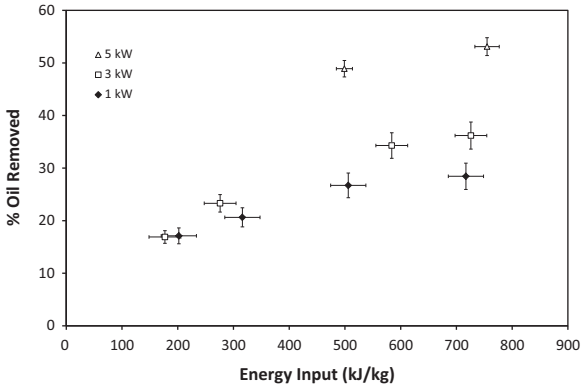

Fig. 8. Effect of energy input and power level on oil removed during microwave processing of 120 g LG Oil Sands in the single mode cavity.

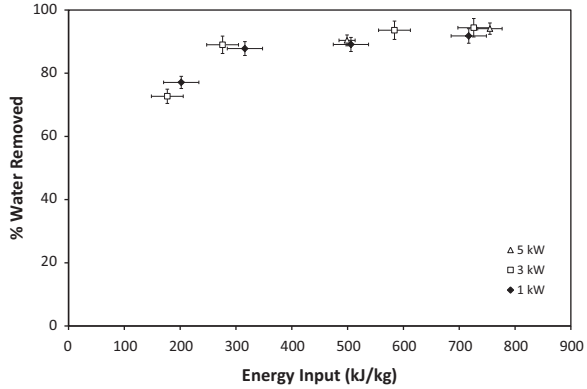

Fig. 9. Effect of energy input and power level on water removed during microwave processing of 120 g LG Oil Sands in the single mode cavity.

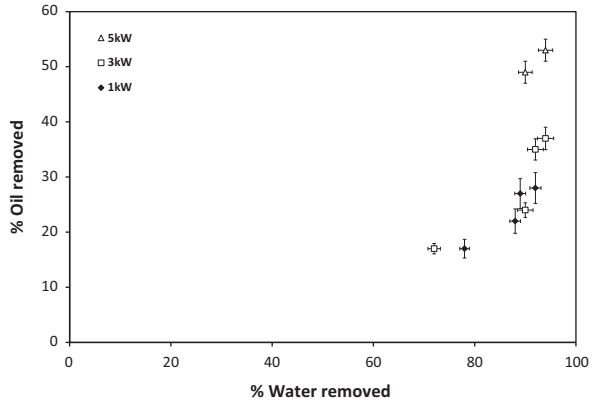

Fig. 10. Relationship between oil and water removal at different power levels during microwave processing of 120 g LG Oil Sands in the single mode cavity.

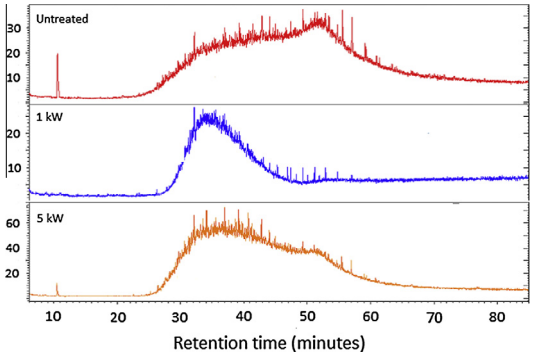

Fig. 11. GC analysis of hydrocarbon liquid collected in the condensation traps after microwave treatment of 120 g LG Oil Sands in the single mode cavity (energy input ~500 kJ/kg), and the oil extracted from untreated LG Oil Sand by solvent extraction.

The data shown in Figs. 4–10 highlight the key role that clay minerals must play if microwave heating is to be used to process Oil Sands. When no clays are present, as with High Grade Oil Sands, no significant heating takes place because the little water that is present occurs as free water, and does not effect removal of the oil phase. When clay minerals are present in Low Grade Oil Sands then higher temperatures can be attained and oil can be removed, in this case up to 53%. Based on the data in this study, the energy requirements are 194 kW h per tonne of process material for 53% oil removal. An important trend shown in this study is that higher power densities lead to greater levels of oil removal at equivalent energy inputs. It is possible that much higher oil yields can be obtained at around 200 kW h/tonne with processing equipment that can safely deliver high power levels, which is much lower than the current 2000–2400 kW h/tonne used by the hot water extraction process [25].

4.6. Oil quality

Oil quality is a parameter that needs to be well understood if microwave processing is to be considered for industrial use. Petroleum fuels for commercial or industrial use are subject to a wide range of tests, including composition, purity, density, toxicity, thermal stability, and miscibility and compatibility with other fluids and materials [26]. Although carrying out the full suite of tests is beyond the scope of this work, attempts were made to get a preliminary indication of the oil quality. American Petroleum Institute (API) gravity is a good initial gauge of oil quality, as crude prices are often posted against degrees API [26]. In this case a large proportion of the extracted oil was condensed within the reactor and pipework prior to the condenser, which is a unique feature of microwave heating since the surrounding environment is cold. Large volumes of solvent were required to collect the adhered oil, and the subsequent separation of oil from solvent resulted in the removal of the lighter components of the oil, and consequently affected the API gravity readings. In order to carry out these measurements, the apparatus would have to be modified to include a vapour recovery system for the cold surroundings present during microwave heating, as this would enable the collection of a large enough oil sample without the need for solvent. For the purposes of the current study Gas Chromatography (GC) was used as the solvent peak could be eliminated from the Chromatogram, and this data is shown in Fig. 11.

The GC data indicates the relative hydrocarbon composition between the different samples. Smaller hydrocarbon compounds pass through the column relatively quickly, and are detected at short retention times. Likewise, larger compounds are less mobile and take longer to pass through the column. Whilst quantification is not possible for the complex mixtures studied here, comparison of the gas chromatograms of the different oil samples clearly shows differences in the distribution of retention times, and hence qualitative differences in composition. The oil from the untreated Oil Sand showed a broad distribution of retention times between 25 and 60 min. The oil collected from the condensation trap during the 1 kW microwave treatment showed a narrower distribution, between 25 min and 50 min, with a clear peak at 33 min. The oil collected at 5 kW showed a broad distribution across a similar range to the untreated sample, but the ratio of light oils to heavy oils is clearly higher than in the untreated sample. This apparent upgrading could be caused by preferential removal of the lighter hydrocarbons, thermal cracking, or reactions between the hydrocarbons and the steam. The data in Fig. 11 suggests that the upgrading effect was greater in the lower power treatment. However, Fig. 8 shows that around twice as much oil was removed from the sample when treated at 5 kW compared with 1 kW. It is therefore more likely a combination of higher local temperatures and greater oil removal that leads to the broader peak of the oil collected at 5 kW. If the upgrading effect was caused by cracking, the effect would be expected to be greater in the higher power treatment owing to the higher local temperatures achieved at higher powers. It is possible that the steam generated during treatment could react with the hydrocarbons, and that could also lead to a modification of the oil composition. Future work will investigate these questions, but based on the evidence presented, the oil upgrading observed here is most likely dominated by the preferential removal of the lighter species.

5. Implications for industrial processing

The aim of this study was to evaluate the potential of microwave heating as a dry process for extraction of the oil phase from Oil Sands. It has been categorically shown that Heavy Grade Oil Sands are not suitable for microwave processing due to their very low water content and the nature of this water. HG Oil Sands are currently used as feedstocks in hot-water extraction processes, and there are no advantages or rationale for using microwaves to treat these materials. Low Grade Oil Sands are more challenging to process using conventional techniques due to the presence of hydrophilic clay minerals, which contribute significantly to the tailings that result from the hot water extraction process. This study has shown that LG Oil Sands are amenable to microwave heating, and that the oil phase can be removed and recovered without the need for water or solvents. The oil is fractionated as

it is removed, leaving a heavy hydrocarbon residue remaining in the solid and a lighter product oil as characterised by GC analysis. 53% of the oil phase was removed in this study at an energy input of around 200 kW h/tonne. Conventional processes utilise 2000–2400 kW h/tonne based on natural gas usage alone [25]. It has been shown that the efficiency of oil removal can be increased by increasing the power density, so further gains in oil removal can be achieved at the same overall energy input. The oil removal was limited by the batch processing employed in this study, and further gains in oil removal are only likely to be observed in a high power density continuous processing system.

There are many options for using high power microwave heating in continuous processing at industrial scale that meet the requirements of this application. The frequency used in this study, 2.45 GHz, is not suitable for industrial processing, so the next stage must be carried out within the 896–922 MHz band, where higher power industrial processing equipment is available. The decision on whether to develop the technology to the next stage will depend on economics. A preliminary economic analysis, based on 2011 Canadian electricity prices [27], suggests that at the current process efficiency of 200 kW h/tonne to remove 53% oil the electricity cost per barrel would be around US$45–50/bbl of oil produced. Given that total operating costs for a surface Oil Sand mining operation are around US$25–35/bbl of sweet synthetic crude oil (SCO) [28], the cost appears prohibitive. However, the efficiency could conceivably be improved to 100 kW h/tonne to extract 80% of the oil, which would result in electricity costs closer to US$15/bbl. Water usage is virtually eliminated in this process, which has significant environmental benefits and reduced costs. The facility would also be able to process a larger proportion of the mined Oil Sands, reducing the economic and environmental impact of the tailings pond. These savings would be offset to some extent by the relatively high cost of microwave hardware. Overall, the results presented here indicate that this technology may become economically viable with an optimised process, and that there is significant potential for reducing the environmental impact of the Oil Sands processing facility.

Further work is required to assess the quality of the product oil obtained. Whilst the preliminary analysis carried out in this study showed that the oil quality was favourable, large quantities of oil from a pilot-scale microwave heating process would be required to carry out the rigorous assessment needed to understand the requirements for further upgrading of the oil. Likewise, a pilot scale process will be required to assess the residual solids and the environmental impact of any remaining hydrocarbons.

The LG Oil Sands from Athabasca that were used in this study had a clay content of around 7%. Feedstocks with similar or higher clay contents are likely to be amenable to microwave processing, with the advantages over conventional processes as outlined above. Feedstock suitability will reduce as the clay and water content decreases, and there is likely to be a threshold below which the Oil Sands will not be suitable.

6. Conclusions

This is the first study to show a fundamental difference in microwave heating behaviour between High Grade and Low Grade Oil Sands, and to attribute this to the location and nature of water within the matrix. Water is the only microwave-absorbing phase within Oil Sands, and oil removal occurs indirectly as a result of selective heating of water. HG Oil Sands contain only fee water, and in very low amounts. The maximum bulk temperature that can be achieved for HG Oil Sands is 70 °C, which leads to negligible levels of oil removal. LG Oil Sands contain clay fines and a combination of free and bound water. This allows temperatures in excess of 250 °C to be reached and the oil to be removed as vapour. The energy requirements are 200 kW h/tonne for 53% oil removal, and oil removal can be increased at the same energy input if a higher power density is used. No water needs to be added to the process, with the heating mechanisms solely dependent on the water that exists within the Oil Sands. Preliminary GC analysis of the recovered oil showed that some upgrading occurred during the process, with lighter hydrocarbons more dominant in the recovered liquid than in the unprocessed Oil Sands. Further work in the form of a continuous processing system is required to assess and optimise the oil yield and quality, nonetheless the findings of this study will allow a technical, economic and environmental case to be made for microwave heating against conventional technologies.

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